CT Associates, Inc.
7121 Shady Oak Road, Eden Prairie, MN 55344-3516
Telephone: (952) 470-0166 Fax: (952) 942-0293
Website: http://www.ctassociatesinc.com

Effect of a Levitronix BPS-4 Pump System on the Particle Size Distribution of Hitachi HS-8005-D4 Slurry

Submitted to:

Reto Schoeb
Levitronix

Submitted by:

Mark Litchy
CT Associates, Inc.

December 18, 2008

Introduction

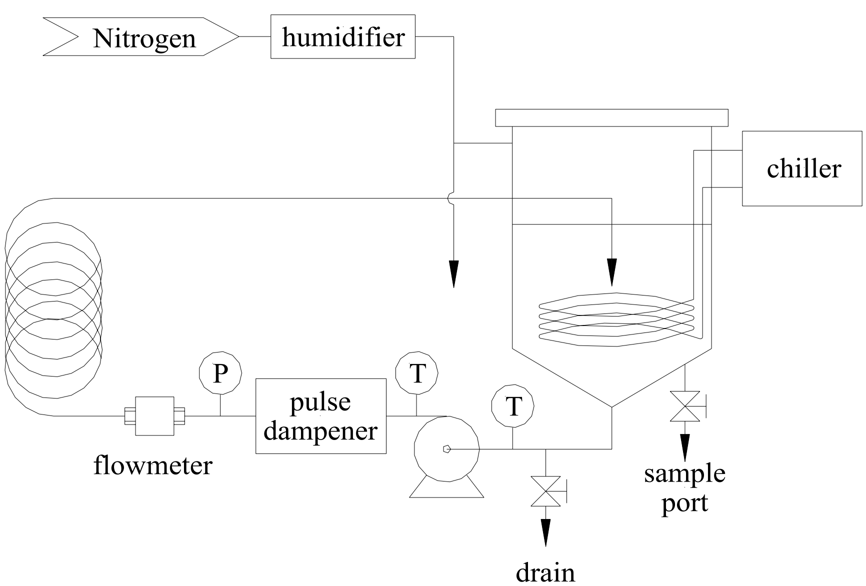
Delivery systems often supply the slurry used to planarize wafers during semiconductor chip manufacturing. These systems pressurize the slurry to deliver it to the tools and circulate it to help keep the particles in suspension. Pressurization and circulation are accomplished by various means including a variety of pump types. Typically, the slurry passes through the equipment providing the motive force approximately 100 times before it is used to polish wafers, i.e. the slurry is "turned-over" approximately 100 times [1]. Some CMP slurries may be damaged due to the mechanical handling of the slurry. For example, particle agglomerates may form which can limit the life of filters or even reduce yield by causing wafer defects.

Previous experiments have shown significant increases in the large particle concentrations when certain types of pumps (diaphragm and bellows) were used to circulate slurry, while Levitronix magnetically levitated centrifugal pumps have not [2]. This test was performed to determine the effect of circulating Hitachi Chemical HS-8005-D4 (lot #: D4E0431) ceria slurry with a Levitronix BPS-4 pump on the health of the slurry. During this test, a number of slurry properties were monitored including the size distribution of the particles in the slurry. No significant changes were observed in any of the slurry properties measured during the test.

Experimental

A Levitronix BPS-4 pump was used to circulate 12.5 liters of slurry at a flow rate of approximately 30 lpm (7.9 gpm) and outlet pressure of 30 psig (2.1 bar). Settling of the slurry in the tank was minimized by drawing from the bottom of a conical bottom tank and by turning the volume of slurry in the tank over in less than 30 seconds. The return line to the slurry tank was submerged below the liquid level of the slurry to avoid entraining gas into the slurry. The return line was also positioned to minimize the formation of a large vortex in the tank that may entrain gas into the slurry. No valves or orifices were used to generate backpressure at the outlet of the pump. Instead, a long length of ½" PFA tubing was used to gradually reduce the pressure at the pump outlet to ambient pressure at the end of the return line to the tank. The speed of the BPS-4 pump was adjusted to 5900 rpm to achieve the desired flow rate and pressure. In each test, the slurry was circulated until more than 1,000 tank turnovers were achieved. The test system was constructed of PFA, except for the conical bottom tank that was constructed of polyethylene.

Figure 1. Test system schematic



Prior to the start of the test, the slurry was redispersed by mixing the slurry with a 3” diameter-folding propeller positioned so that it was within 1 cm of the bottom of the 10L container. The propeller was operated at a speed of approximately 300 rpm for 60 minutes. The container was periodically rotated during mixing. After mixing, the bottom of the container was checked to ensure that there was no sediment remaining. The slurry was then diluted 1:1 by weight with ultra pure water by slowly adding water while continuously mixing the slurry. Hitachi Chemical provided these mixing recommendations.

The tank holding the slurry was blanketed with nitrogen to prevent absorption of carbon dioxide from the air which can change the pH of the slurry. The nitrogen was humidified to prevent dehydration of the slurry. Shifts in the pH and dehydration can both result in slurry particle agglomeration. The relative humidity in the tank was > 90% throughout the test. A chiller and stainless steel coil were used to maintain the slurry at 20 ± 2°C during the test.

Samples were drawn from the system at selected times for analysis. Both the large particle tail and the working particle size distributions (PSDs) were measured. The large particle tail PSD was measured using a Particle Sizing Systems AccuSizer 780 optical particle counter. The AccuSizer uses a combination of light scattering and light extinction to measure the size distribution of particles ≥ 0.56 µm. The slurry samples were diluted approximately 5,000,000:1 with filtered deionized water prior to measurement of the PSD. No information is reported for particle sizes ≥ 1.5 µm since the PSD was steep and the dilution ratio required for these measurements was so high that the concentration of particles ≥ 1.5 µm was below the detection limit of the test system (~10^7 particles/ml). Attempts made to measure concentrations for particle sizes larger than 1.5 µm at lower dilution ratios using extinction mode only were unsuccessful due to the high concentration of particles in this slurry. Between samples, the entire system was thoroughly flushed with deionized water. Data from selected particle size channels were analyzed.

The size of the working particles was measured using two techniques. In the first technique the slurry was diluted 1000:1 into filtered deionized water, the particles in the diluted slurry were dispersed into air using ultrafine atomization (UFA) and the size and concentration of the particles was measured using a scanning mobility particle sizer (SMPS) [3]. The working particle PSD was also measured using a Particle Sizing Systems NICOMP 380ZLS (Santa Barbara, CA) that determines particle size by dynamic light scattering. The particle zeta potential was also measured using this instrument. The zeta potential samples were diluted approximately 75:1 into deionized water while the size distribution samples were diluted approximately 600:1 into deionized water. All measurements were performed at a temperature of 23°C. Each PSD measurement was made over 10 minutes while each zeta potential measurement was made over 2 minutes. The PSD and zeta potential measurements of each sample were performed in triplicate and quintuplicate, respectively. The size measurement data were analyzed using the instrument’s Gaussian distribution assumption.

Other slurry health parameters measured included percent total solids, specific gravity, and pH. Measurements of each sample were performed in triplicate.

Results and Discussion

Figure 2 shows the cumulative PSDs of the slurry large particle tail. The initial PSD, measured prior to the start of each test, is presented as well as PSDs after selected numbers of turnovers.

Figure 2. Cumulative PSDs of the large particle tail measured during pump tests

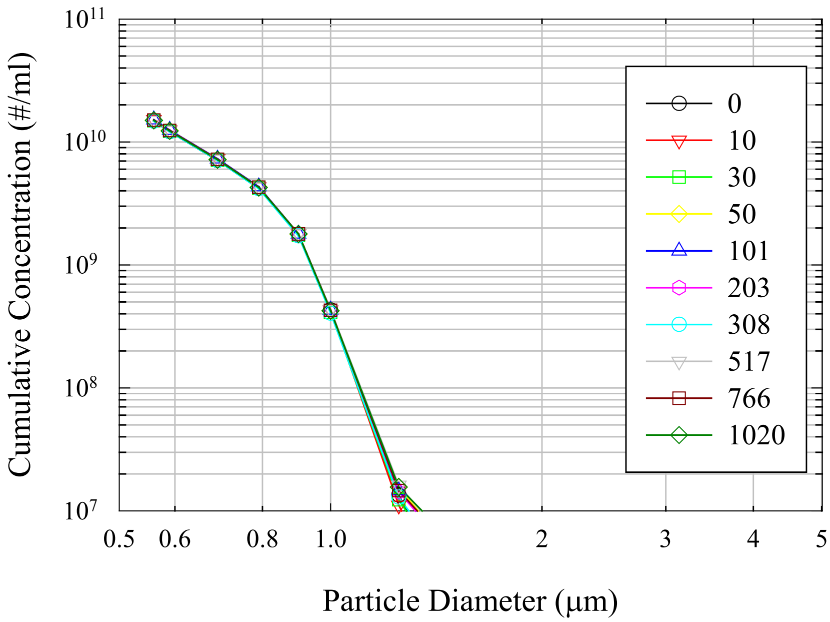
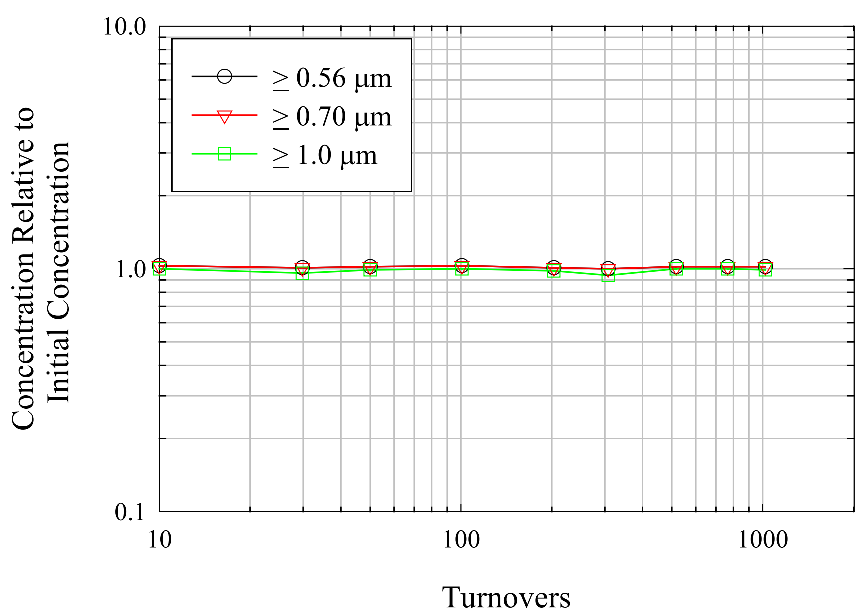


Figure 3 presents the ratios of particle concentrations at each test point to the particle concentration at the start of the test. The concentration ratios for selected size channels are presented as a function of tank turnovers.

Figure 3. Particle concentrations relative to the initial concentration for selected size channels



Figures 2 and 3 indicate that there were minimal changes in the large particle tail of the slurry PSD during all the three pump tests, although there may have been changes at particles sizes ≥ 1.5 µm, which we were unable to measure.

Table I shows a summary of the relative particle concentration changes during each test after approximately 100 and 1,000 turnovers for selected particle sizes. These points were chosen since slurry is typically turned over on the order of 100 times prior to use, while 1,000 turnovers is believed to be a conservative upper estimate in most slurry delivery systems.

Table I. Summary of the relative particle concentrations changes for selected times and particle sizes

Particle Concentrations Relative to the Initial Particle Concentrations		
Particle Size	100 Turnovers	1,000 Turnovers
≥ 0.56 μm	1.0	1.0
≥ 0.7 μm	1.0	1.0
≥ 1.0 μm	1.0	1.0

Figures 4 and 5 present working particle size distributions measured using the UFA/SMPS technique. Figure 4 presents number-weighted distributions, while Figure 5 presents volume-weighted distributions. The graphs indicate that the distribution has a single mode with a number-weighted peak at approximately 80 nm and a volume-weighted peak at approximately 130 nm.

Figures 4 and 5 indicate that there was little change in either the number-weighted or the volume-weighted PSDs during the test. This is shown further in Figure 6, which presents cumulative number concentrations of selected particle sizes as a function of turnovers.

Figure 4. Differential number-weighted working particle PSDs

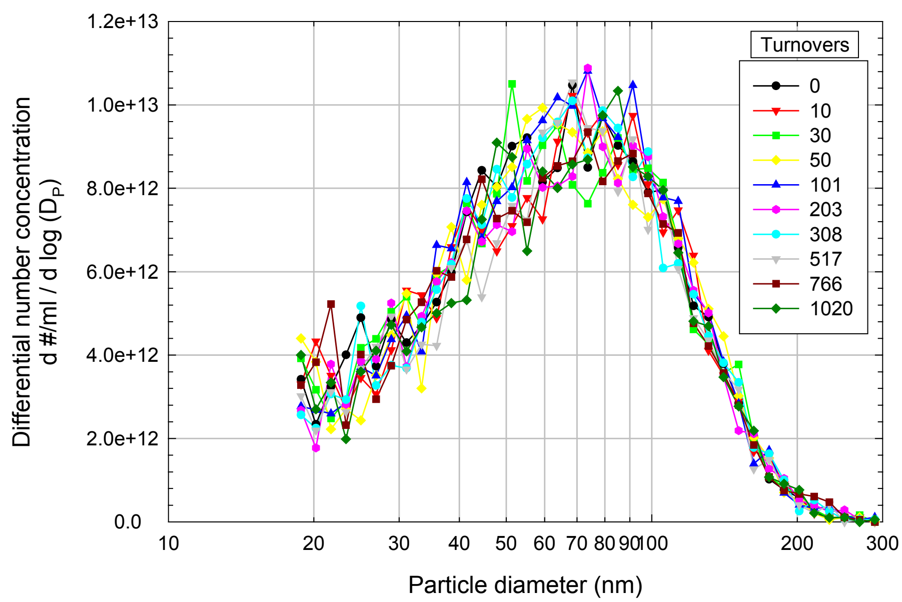


Figure 7 presents the volume-weighted mean and 99th percentile particle diameters (99% of the particles have diameters less than this size) of the working PSD measured by dynamic light scattering as a function of tank turnovers. Error bars are included and represent ± 3 standard deviations. (The initial values were included on the plot at 1 turnover.) The volume-weighted mean and 99th percentile particle diameters at the start of the test were 191 and 445 nm, respectively. The mean size of 191 nm agrees qualitatively with the volume-weighted size distributions measured using UFA/SMPS (Figure 5). Also included in Figure 7 were the zeta potential measurements taken during the test. The zeta potential of this slurry was approximately -27 mV. No significant changes in the working PSD or zeta potential were observed during the test.

Figure 5. Differential volume-weighted working particle PSDs

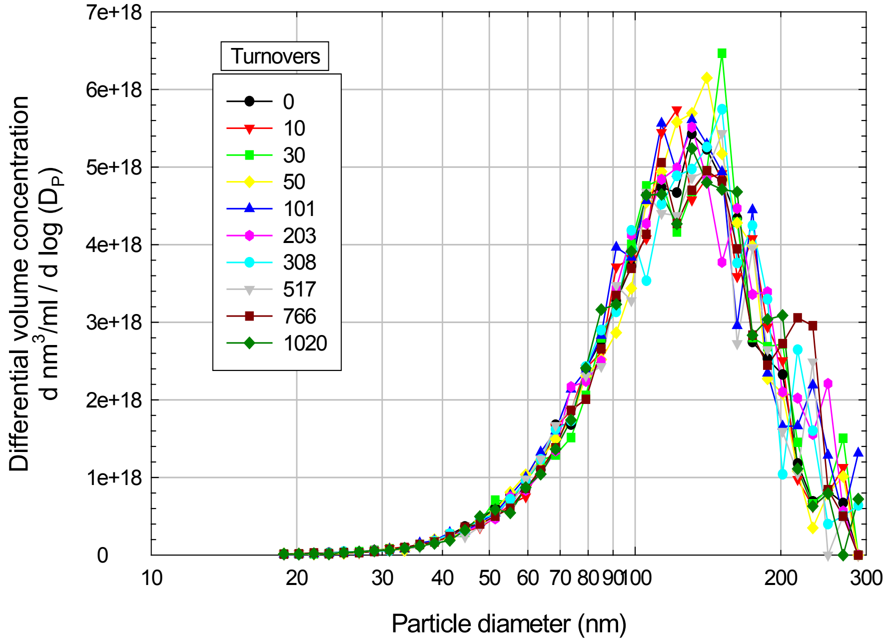


Figure 6. Cumulative working particle concentrations as a function of turnovers

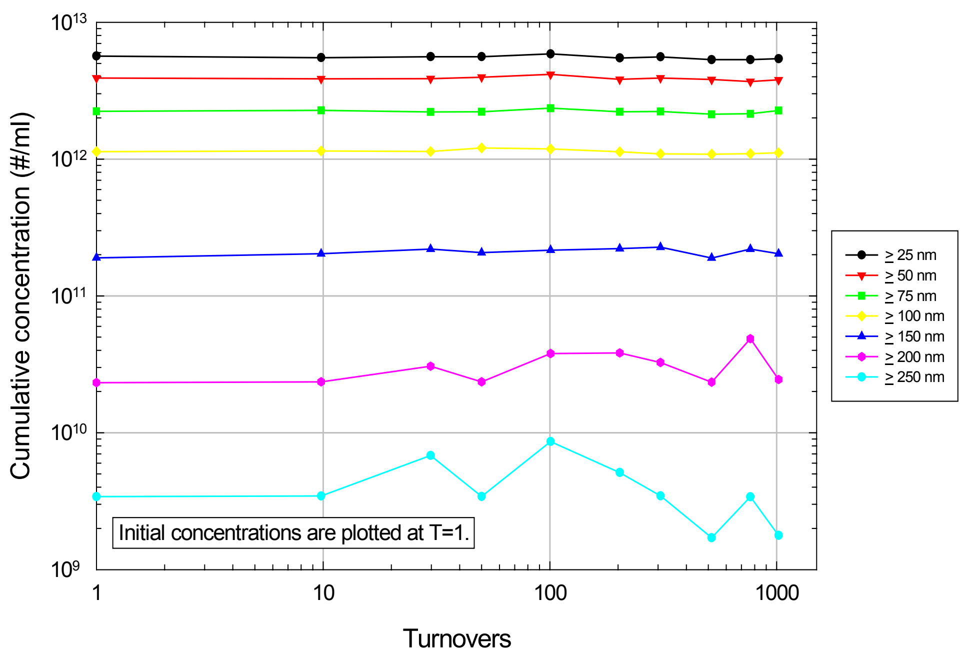


Figure 8 presents the total percent solids, specific gravity, and pH as a function of tank turnovers. Error bars are included in each graph and represent ± 3 standard deviations. (The initial values were included on the plot at 1 turnover.) No changes in these slurry properties were observed during the test.

Figure 7. Working particle size and zeta potential measurements as a function of turnovers

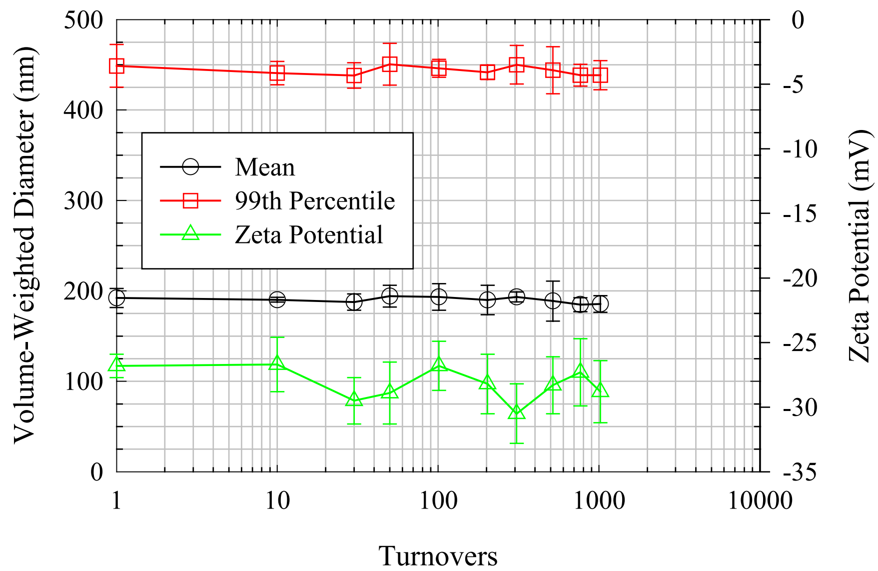
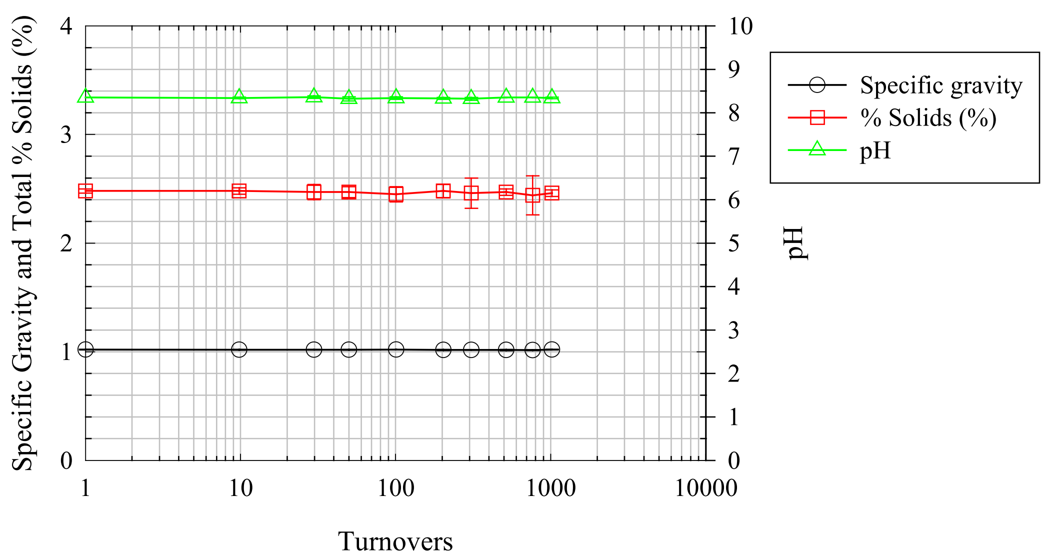


Figure 8. Total % solids, specific gravity, and pH measurements as a function of turnovers



Summary

A Levitronix BPS-4 centrifugal pump was tested to determine how its use affected the size distribution of particles in Hitachi Chemical HS-8005-D4 slurry. The slurry was circulated until it had passed through the pump approximately 1,000 times.

No significant changes were observed in any of the slurry properties measured (large particle tail, working PSD, total percent solids, pH, density, or zeta potential) during the pump test. Note that due to the high concentration of particles in this slurry, no concentration information is presented for particle sizes ≥ 1.5 µm.

References

1. Personnel communication with J. Kvalheim, BOC Edwards Chemical Management Division, Chanhassen, MN, March, 2003.
2. Litchy MR and DC Grant (2007). "Effect of pump type on the health of various CMP slurries", *Semiconductor Fabtech*, 33rd Edition, pp 53-59.
3. Grant DC (2008). "A New Method for Determining the Size Distribution of the Working Particles in CMP Slurries," presented at the 2008 CMP Users Conference, sponsored by Levitronix.